GCE

Rewarding Learning

Revised GCE

Chemistry

A2 Practical Support Document

For first teaching from September 2016

A2 Practical Support Document

1 Carrying out experiments to determine the rate of a reaction using a variety of methods to determine the concentration of reactants and/or products

Different methods which may be used include:

(a) Using a colorimeter – this is used if the reactant or product is coloured

- prepare a range of samples of different concentrations of the required solutions
- calibrate the colorimeter using the appropriate filter and a blank solution
- record the absorbance of each solution of known concentration
- draw a calibration curve of absorbance against concentration
- place the reaction mixture in the colorimeter and record the absorbance at different times
- use the calibration curve to convert absorbance to concentration and plot a graph of concentration against time. A gradient of a tangent at any concentration on the graph gives the rate at that concentration.

(b) Titrating

- the reaction is started by mixing the solutions and a sample is removed by pipette at various times
- quench the sample to stop the reaction by rapid cooling/adding a large volume of water/adding a chemical to remove a reactant which is not being monitored
- titrate the sample to find the concentration of the reactant or product
- plot a graph of concentration against time; the gradient of a tangent at any concentration on the graph gives the rate at that concentration.

  For example, to determine the rate of an esterification reaction the samples are titrated with standard alkali. To determine the rate of a reaction involving iodine, the remaining unreacted iodine is titrated with standard sodium thiosulfate solution until straw/yellow, then starch indicator is added and the titration continued until the starch changes from blue-black to colourless.

(c) Measuring pH over time - this is used if H^+ or OH^- ions are a reactant or a product

- start the reaction
- record the pH at different times using a pH meter
- calculate the hydrogen ion concentration using $pH = -\log_{10}[H^+]$
- a graph of $[H^+]$ against time can be drawn and again gradients of tangents at various $[H^+]$ can be taken, which equal the rate at each point.

(d) Measuring the volume of a gaseous product over time, can be used if the product is a gas

- attach a gas syringe to a sealed reaction vessel, or collect the gas over water in an inverted measuring cylinder/burette
- start the reaction
- record the volume of gas produced at different time intervals
- a graph of volume of gas against time can be plotted.

(e) Measuring the mass of a gaseous product, over time, can be used if the product is a gas

- a flask containing one reactant is placed in a flask on a balance and the mass recorded
- add the second reactant and start the clock
- record the mass at different time intervals until the reaction is finished
- a graph of mass against time is plotted

2 Making buffer solutions from calculated quantities of salts and acids and determine their pH values using Universal Indicator (UI) paper and a pH meter

- use a burette to measure out calculated volumes of equimolar solutions of a weak acid and the salt of the weak acid and add to a beaker
- mix (use a magnetic stirrer)
- record the pH of the prepared buffer using Universal Indicator paper and a pH meter.

3 Determining the shape of a titration curve by measuring the pH using specialised pH paper or a pH meter for the titration of an acid with a base

- place acid/base solution of known volume and concentration in a conical flask.
- add 3 drops of an appropriate indicator
- record the pH of the solution using a pH meter or narrow range pH paper
- fill a burette with acid/base of known concentration.
- add the acid/base in 5 cm^3 portions from a burette, mixing with a magnetic stirrer
- record the pH, after each addition
- add the acid/base in 1 cm^3 portions as the end point approaches
- continue to add two 5 cm^3 portions after the end point
- plot a graph of pH against volume of solution added (titration curve)

4 Determining the pH of a variety of salts using pH paper or a pH meter to illustrate the relative strengths of acids and bases

- prepare solutions of known concentration of a variety of salts
- record the pH using a pH meter or narrow range pH paper.

5 Preparing, recrystallising and determining the melting points of 2,4-dinitrophenylhydrazones; laboratory preparation of 2,4-dinitrophenylhydrazones

- place 5 cm^3 of 2,4-dinitrophenylhydrazine solution in a suitable container.
- add some drops of the test liquid e.g. propanal or the test solid dissolved in ethanol.
- if crystals do not form add some dilute sulfuric acid and warm the mixture.
- cool the mixture in iced water.
- filter off the crystals using suction filtration.
- recrystallise. - dissolve the impure crystals in the minimum volume of hot solvent. Filter when hot by gravity filtration, using a hot funnel, or fluted filter paper, to remove insoluble impurities. Allow filtrate to cool and crystallise
- filter off the crystals using suction filtration
- dry by sucking air over the crystals in the Buchner and then in a low temperature oven/dessicator
- determine the melting point.

Suction filtration method

- Place filter paper in a Buchner funnel.
- Place Buchner funnel in a Buchner flask.
- Attach the flask to a suction pump and suck air through the flask.

Diagram of suction filtration should include:

- Buchner funnel and filter paper.
- Buchner flask.
- apparatus complete with no gaps.
- suction indicated.

Melting point method

- place some solid in a capillary tube/melting point tube sealed at one end
- heat slowly (using melting point apparatus)
- record the temperature at which the solid starts and finishes melting
- repeat and average the temperatures
- compare the temperature(s) with known values in a data book

6 Using Fehling's solution and Tollens' reagent to distinguish between aldehydes and ketones

- add a few drops of the unknown solution to 1 cm^3 of freshly prepared Tollens' reagent in a clean test tube.
- warm in a hot water bath.
- freshly prepare Fehling's solution by mixing 1 cm^3 of Fehling's solution A with 1 cm^3 of Fehling's solution B.
- add a few drops of the unknown solution to 1 cm^3 of freshly prepared Fehling's solution reagent in a test tube. Warm in a hot water bath

7 Preparing a carboxylic acid from an alcohol

- add concentrated sulfuric acid to water in a pear shaped/round bottomed flask.
- swirl the solution and cool the flask to dissipate the heat and to prevent spitting
- add potassium dichromate(VI) solution and swirl the mixture.
- add anti-bumping granules.
- attach a vertical condenser.
- add the alcohol slowly, down the condenser, to the acidified potassium dichromate(VI) solution, cooling the reaction flask in a water bath.
- heat the mixture under reflux.
- distil off the acid.

For information on reflux and distillation, see AS practical document.

8 Carrying out test tube reactions of a carboxylic acid with sodium carbonate, sodium hydroxide and aqueous ammonia and measure the pH changes

- place 1 cm^3 of the carboxylic acid in a boiling tube
- record the pH using pH paper
- add a spatula measure of sodium carbonate and record observations
- record the pH using pH paper

- repeat the experiment using 1 cm^3 of sodium hydroxide solution instead of sodium carbonate
- repeat the experiment using 1 cm^3 of aqueous ammonia instead of sodium carbonate.

9 Preparing a liquid ester from a carboxylic acid and an alcohol

- place a mixture of the alcohol and concentrated sulfuric acid in a pear shaped/round bottomed flask.
- add a mixture of the alcohol and carboxylic acid slowly from a dropping funnel.
- swirl the mixture
- add some anti-bumping granules and heat under reflux
- arrange the apparatus for distillation, heat the mixture gently and collect the distillate around 2° C either side of the boiling point of the ester
- place the crude ester in a separating funnel and shake with sodium carbonate solution. Invert the funnel and open the tap occasionally to release pressure .Remove the stopper, allow the layers to separate and discard the aqueous layer; (to decide which layer is the aqueous one, add 5 cm^3 of water to the separating funnel, and the aqueous layer is the one which increases in volume)
- add a spatula measure of anhydrous calcium chloride/sodium sulfate/magnesium sulfate (drying agent) to the organic layer in a flask, stopper and shake. Repeat until the ester changes from cloudy to clear.
- filter or decant to remove calcium chloride.
- redistill to remove any remaining organic impurities, collecting the fraction at the boiling point.

  Note that for some small esters such as ethyl ethanoate, distillation can be used rather than reflux - the ester can be distilled off as soon as it is formed as it has the lowest boiling point of the substances present.

10 Preparation of methyl 3-nitrobenzoate

- dissolve some of methyl benzoate in some concentrated sulfuric acid.
- cool the solution in ice.
- prepare the nitrating mixture by carefully adding concentrated sulfuric acid to concentrated nitric acid and then cool this mixture in ice.
- add the nitrating mixture drop by drop to the solution of the methylbenzoate, stirring with a thermometer and keeping the temperature below 10 °C.
- when the addition is complete allow the mixture to stand at room temperature for 15 minutes.
- pour the reaction mixture onto crushed ice and stir until all the ice has melted and crystalline methyl 3-nitrobenzoate is formed.
- filter the crystals using Buchner filtration, wash with cold water, recrystallise from ethanol, filter off and dry

11 Titrating iodine with sodium thiosulfate solution using starch and hence estimate oxidising agents by their reaction with excess acidified potassium iodide

- rinse a pipette with potassium iodate(V) solution and using a pipette filler, pipette 25.0 cm^3 of standard potassium iodate(V) solution into a conical flask
- add approximately 1.5 g of potassium iodide (excess) and excess of sulfuric acid (by measuring cylinder).

- rinse the burette with the standard sodium thiosulfate, and then fill the burette with sodium thiosulfate, making sure the tap is filled.
- add standard sodium thiosulfate solution from the burette until the solution is straw/yellow in colour and add 3 drops of starch indicator solution.
- titrate until the indicator changes colour
- repeat until results are concordant (within 0.1 cm^3)

If hydrogen peroxide is used as an oxidising agent, instead of the forst step, pipette 25.0 cm^3 of hydrogen peroxide into a conical flask; add a few drops of ammonium molybdate catalyst and proceed as above.

12 Titrating acidified potassium manganate(VII) solution with reducing agents

- rinse the pipette with a solution of a reducing agent and use the pipette and safety filler to pipette 25.0 cm^3 into a conical flask.
- rinse the burette with a standard solution of potassium manganate(VII) and then fill the burette ensuring the tap is filled.
- add excess sulfuric acid using a measuring cylinder, to the conical flask
- add potassium manganate(VII) solution from the burette into the conical flask and swirl until the solution turns from colourless to pink
- repeat until results are concordant (within 0.1 cm^3)

13 Determining the purity of a Group II metal oxide or carbonate by back titration

- weigh out a mass of the solid (Group II metal, Group II oxide or Group II carbonate)
- add an excess of hydrochloric acid
- add the solution to a 250 cm^3 volumetric flask and make up with deionised water until the bottom of the meniscus is on the mark
- rinse a pipette with the solution from the volumetric flask, and use the pipette and safety pipette filler to pipette 25.0 cm^3 into a conical flask.
- add 3 drops of phenolphthalein indicator
- rinse a burette with standard alkali solution, and fill the burette
- add alkali from the burette, to the conical flask until the indicator changes colour
- repeat until results are concordant (within 0.1 cm^3)

14 Carrying out paper and thin-layer chromatography and measure the R_f values of the components and interpret the chromatograms

- draw a base line using a pencil close to the bottom of the paper/thin-layer plate
- spot the samples onto the paper/thin-layer plate using a capillary tube. Allow the spots to dry. Repeat to make the spots concentrated.
- place the paper in the tank containing a shallow amount of solvent, cover with a lid, and allow the solvent to run up over the spots until the solvent almost reaches the top of the paper/thin layer plate. Mark the solvent front. Allow to dry.
- if the substances to be separated are colourless, the spots are made visible by spraying with a locating agent e.g. ninhydrin
- measure the distance travelled by the solvent front, and the distance moved by each spot (to centre of spot) and calculate the retardation factor, R_f.

Two-way chromatography

- carry out the method as above
- rotate the paper/thin-layer plate through 90°
- run in a second solvent.- place the paper in the tank containing a shallow amount of a different solvent, and allow the solvent to run up over the spots until the solvent reaches the top of the paper. Mark the solvent front
- if the substances to be separated are colourless, the spots are made visible by spraying with a locating agent e.g. ninhydrin
- measure the distance travelled by the solvent front, and the distance moved by each spot (to centre of spot) and calculate the retardation factor, R_f for this solvent.

15 Using ethylene diamine, phenylamine and aqueous ammonia to demonstrate ligand replacement based on lone pair availability

- place 2 cm^3 of a metal ion solution in a boiling tube
- add 1 cm^3 of phenylamine, shake and record observations
- add 1 cm^3 of aqueous ammonia solution, shake and record observations
- add 1 cm^3 of ethylene diamine, shake and record observations.

16 Demonstrating the relative strengths of ligands using hexaaquacopper(II) ions in solution and hydrochloric acid

- add 2 cm^3 of a hexaaquacopper(II) ion solution to a test tube
- add 2 cm^3 of concentrated hydrochloric acid and record observations.

17 Carrying out qualitative detection tests for the formation of transition metal hydroxides with sodium hydroxide solution and aqueous ammonia

- add 2 cm^3 of a solution of the transition metal ion to a test tube
- add a few drops of aqueous ammonia and record observations
- add excess (5 cm^3) of aqueous ammonia and record observations
- repeat using sodium hydroxide solution instead of aqueous ammonia.

18 Carrying out the reduction of acidified ammonium vanadate(V) (ammonium metavanadate) with zinc and observing the sequence of colours

- dissolve some ammonium vanadate(V) (ammonium metavanadate) in hydrochloric acid in a conical flask
- add two spatulas of zinc as a reducing agent
- stopper the flask with cotton wool, to allow the hydrogen to escape, and slow down the entry of air.
- observe the sequence of colours.

19 Determining the electrode potentials of a series of cells and predicting their values using standard electrode potentials

- set up a half-cell - a metal dipping into a 1 M solution of its ions in a beaker

- attach via wires, and a voltmeter to a second half-cell using a different metal and a 1 M solution of its ions
- use a salt bridge to connect the two beakers
- record the voltage.

20 Determining the amount of a carbonate, for example calcium carbonate or magnesium carbonate, in an indigestion tablet

As magnesium hydroxide and calcium carbonate are insoluble in water, their percentage in an indigestion remedy can be determined by back titration (see 13)

21 Preparing aspirin using salicylic acid and ethanoic anhydride

- place 1.0 g of 2-hydroxybenzoic acid in a dry pear shaped flask and add 2 cm^3 of ethanoic anhydride.
- safely add 8 drops of concentrated phosphoric(V) acid
- heat under reflux for 30 minutes.
- add water to hydrolyse any unreacted ethanoic anhydride to ethanoic acid
- pour the mixture onto 400 g of crushed ice in a beaker.
- remove the product by suction filtration,
- recrystallise from water
- dry in a desiccator/low temperature oven
- determine the melting point

22 Using chromatography to compare the purity of laboratory-made aspirin with commercial tablets

- draw a base line using a pencil close to the bottom of the paper/thin-layer plate and draw two pencil crosses on the base line
- place some of the laboratory-made aspirin solid on a watch glass and dissolve in a few drops of solvent such as ethanol. Use a capillary tube to place a spot of the solvent on a pencil cross. Allow the spot to dry and repeat, ensuring the diameter of the spot is no more than 0.5 cm. This produces a concentrated spot. Repeat this for the commercial solid.
- place the paper/thin-layer plate in the tank containing a shallow amount of solvent, cover with a lid and allow the solvent to run up over the spots until the solvent reaches the top of the paper. Mark the solvent front. Allow to dry.
- The spots are made visible by placing the paper/thin-layer plate in a beaker containing iodine crystals.
- measure the distance travelled by the solvent front, and the distance moved by the centre of each spot and calculate the R_f value of each spot

COUNCIL FOR THE **CURRICULUM, EXAMINATIONS** AND **ASSESSMENT**
29 Clarendon Road, Clarendon Dock, Belfast BT1 3BG
Tel: +44 (0)28 9026 1200 Fax: +44 (0)28 9026 1234
Email: info@ccea.org.uk Web: www.ccea.org.uk

Rewarding Learning